

6-1925

A Study of the Equilibrium between Lead, Lead Iodide and Iodine

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Day, William Worden, "A Study of the Equilibrium between Lead, Lead Iodide and Iodine" (1925). *Honors Theses*. 1738.
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A Study of the Equilibrium between Lead,
Lead Iodide and Iodine.

A thesis, presented to the Department of Chemistry of
Union College, in partial fulfillment of the requirements for the
Degree of Bachelor of Science in Chemistry, by

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Charles B. Hurd

May 20, 1925

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A Study of the Equilibrium between Lead, Lead Iodide and Iodine

Introduction

Although considerable work was done in 1924 by N. B. Reynolds¹ on the equilibrium between Lead, Lead Iodide and Iodine, there is, at the present time, no accurate data as to vapor pressure of Iodine over Lead Iodide at various temperatures. The same is likewise true of almost all metal halides and this work was undertaken to get such data as could be computed by direct pressure measurements. The fact that these vapors are very corrosive makes it impossible to use a McLeod gauge or any other common form of manometer to measure the pressure directly.

History

To write a history of the work that has been done on this problem would be merely to duplicate what Reynolds¹ has already very adequately done in his thesis of 1924.

Apparatus

Since the vapor, of which the pressure is to be measured, will react with or dissolve in the liquid of any ordinary manometer, we decided that the only way in which progress could be made was to use a device developed by D.F. Smith and N. W. Taylor². This consists of a thin glass diaphragm which separates the corrosive gas from a secondary chamber whose pressure can be measured with a

manometer. According to Smith and Taylor it is a very simple matter to make these glass diaphragms but we found that it required a large number of trials and failures before good ones could be made. This apparatus is a modification of the glass pressure-measuring device described by Daniels and Bright³ and later modified by Karren, Johnston and Wulf⁴. The new device, as we use it, is made by blowing a fairly thin bulb (B) on the end of a 4 - 8 mm. glass tube and flattening the end of the bulb, but during the flattening, so directing the flame that the diaphragm (D) takes on a slight concave shape when viewed from the outside. Change of pressure then causes a sharp "click" as the diaphragm passes from a concave to a convex position and a second "click" when it returns to its normal concave position. Thus we have two critical positions at which there is a "clicking" noise, once when pressure is directed outward and once when it is inward. These bulbs can be successfully made of ordinary lime glass or of Pyrex glass. It is not necessary that the membrane of glass be excessively thin. They may be strong enough to withstand a pressure difference of one atmosphere and still be sensitive to .15 mm. In general the ones with the largest zero correction give the loudest "click" but it is the glass, rather than the gas, that carries the sound so that they all can easily be heard. We blew sensitive bulbs which ranged from 10 - 25 mm. across the flat portion. The method of mounting is shown in the diagram.

The pressure gauge (P) consists of two columns of mercury with stop-cocks so arranged that the space over one column can be evacuated and then the pressure acting on the other column is equal to the difference in the levels of the mercury in the two columns.

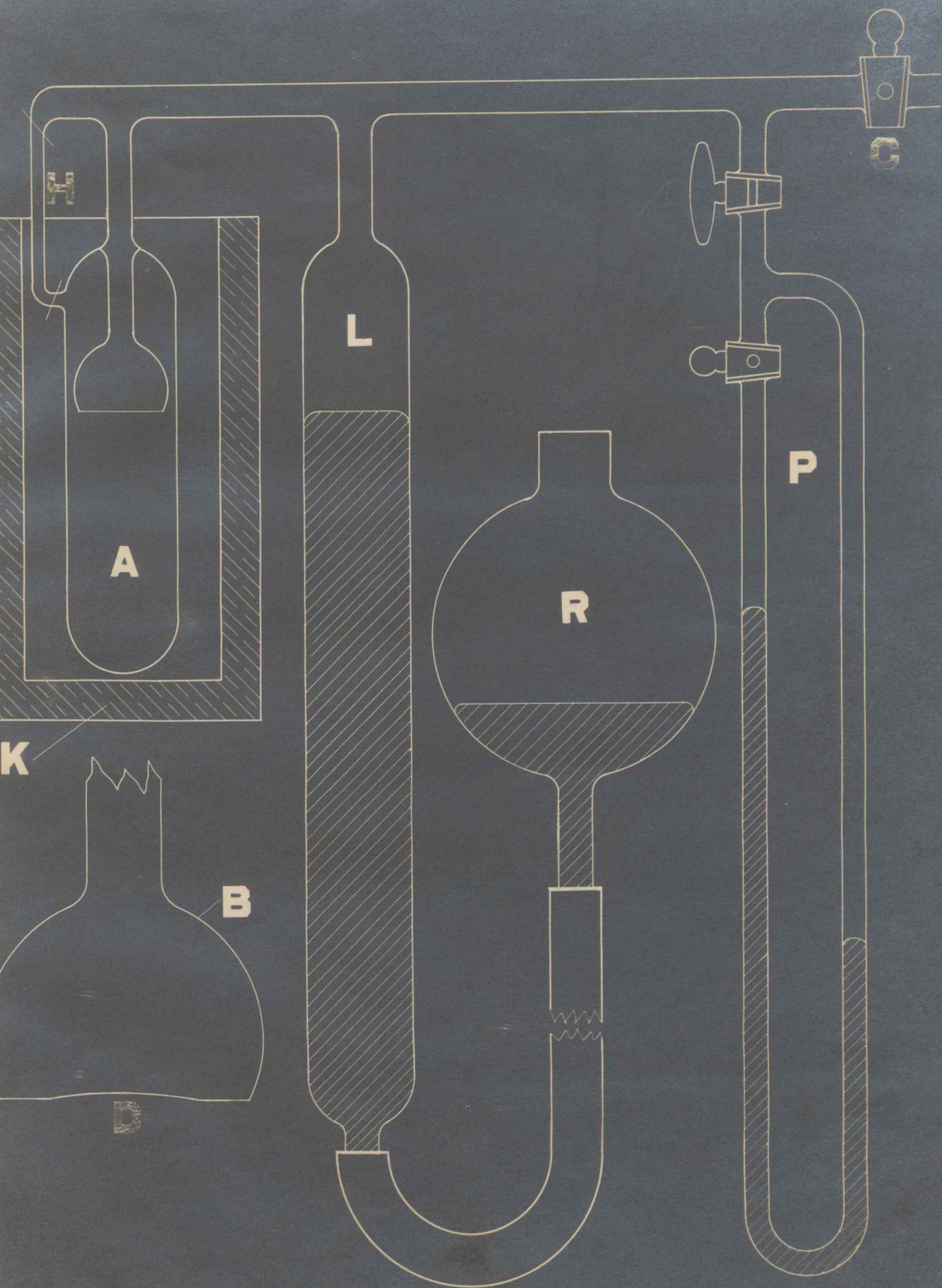
By this method atmospheric pressure does not enter into the computations in any way.

The dissociation chamber (A) consists of a large tube (1" in diam.) closed at the bottom, into which the Lead Iodide is placed. The glass diaphragm is sealed into the top of (A) as shown in the diagram. The dissociation chamber is connected with the rest of the system by a small tube (H) which is sealed off after the system is evacuated and ready to run. The whole dissociation chamber and diaphragm are placed in a tight electric furnace (K) which can be kept at a constant temperature.

The chamber (L) is a tower of fairly large volume which can be filled with mercury to any level by raising or lowering the reservoir (R). Since the volume of this tower is much larger than the total volume of the rest of the system, it is possible, merely by changing amount of mercury in (L), to double or halve the pressure in the secondary system. This is very convenient when we are locating the "clicking" points.

By means of the stop-cock (C) the system can be connected to a high grade vacuum pump which will reduce the pressure to a very low value (1 micron). The complete system is of glass with glass seals in all instances except the flexible connection to the pressure regulating reservoir. The whole system was so free from leaks that after evacuation no change in pressure could be noted over a period of three or four days. The mercury levels in the pressure gauge were read from a very accurate cathetometer which is calibrated down to $1/200$ of a cm.

I experienced a great deal of difficulty in making the apparatus because I am not a glass blower and could not easily



get the services of a professional. It, therefore, took a long time to get the glass ware assembled and then also we would have an accident now and then which would quickly undo or cause a whole week's work. Another source of delay at first part of year was the getting of a vacuum pump. This pump was borrowed from the General Electric Company. Another incidental delay was due to a scarcity of mercury in the laboratory. This was soon overcome by buying some new mercury from the General Electric Company.

The whole period of time has been used in getting apparatus in shape for business and no real work has been done on the problem proper. However, we feel really optimistic about what can be done with the apparatus as it is now assembled.

Results

Table I is a typical run with one of the glass diaphragms and shows the consistancy of the readings (lime glass).

Table I.

<u>Temperature</u>	<u>Actual Pressure</u>	<u>Measured Pressure</u>
25° C	35.0 cm.	35.006 cm.
	30.0	29.993
	26.4	26.401
	20.5	20.485
	16.0	15.990
	7.6	7.608
	1.0	.992
100°	27.5	27.515
200°	19.0	18.988
300°	75.2	75.194

The results of Table I show that the pressures as measured are accurate to .15 mm. and are equally accurate at all temperatures from 25° to 300°.

Summary

An apparatus has been constructed by which we believe it will be possible to make a series of studies of equilibria in systems containing gases which would be corrosive to mercury. The system lead, iodine, lead iodide is to be studied with this set-up.

Bibliography

1. Thesis of 1924 by N. B. Reynolds
2. J.A.C.S. 46, 1393 (1924)
3. J.A.C.S. 42, 1131 (1920)
4. J. Ind. Eng. Chem. 14, 1015 (1922)